2. **Plans:** The menthol counterparts have not been evaluated at this time. However, we will POL test B&H 100's Menthol in May. The cigarettes have been approved by the Richmond Panel and will be shipped soon. This will be the only testing of the menthols.

   We have been testing the feasibility of including NET into Lark, Marlboro Ultra Lights, and Merit Ultima. We will try to conclude this work during the fourth quarter, using a lower priority.

   Saratoga will be made during the third quarter. This was delayed since it has to be made at Stockton Street.

   Two Danchi tests will be run for Parliament. The first has been made, but CI data is not complete. The second will be run at the end of May.

3. **Conclusions:** The 3–component NET product with sugar/glycerin casing and expanded at a 595°F tower temperature allows the higher expanded tobacco inclusion level to be achieved. Based on weight reductions achieved in POL's, 15% NET is approximately equal to 17% BLDET or DIET in volume displacement in the finished cigarette.

4. **Contributors:** C. Moogalian, J. Dobbs, D. Leister, B. Riggan, B. Peace, G. Yatrakis, B. Taylor, T. Clarke, V. Covington, J. Atkinson, P. Grantham, G. Carter, W. Mokarry, G. Romig, G. Inge, Analytical Research, Materials Evaluation Lab, Semiworks Primary and Make Pack Personnel, Chemical Research, Flavor Technology, R. Southwick

III. Objective: Develop Design Criteria for Conversion of DIET Facilities to the NET Process.

A. Strategy:

1. **Results:** An evaluation of low pressure, 430 psig, NET impregnation capacity for both Cabarrus and the MC was completed based on normal cutting OV followed by cylinder drying of the feed tobacco to 15% OV. Results show the following:

   Cabarrus – Maintain 5000 #/hr capacity with 3 impregnators.

   MC – Derate capacity from 4000 #/hr to 3400 #/hr per line based on two impregnators per line.

   It may be possible to maintain MC capacity at 4000 #/hr per line with hot cutting at 16 to 17% OV.

   An Australian report on their evaluation of low pressure Batch NET expanded tobacco shows an improvement in cigarette "physical quality" or "a weight savings opportunity" at higher inclusion levels.

2. **Plans:** Support Manufacturing, both Domestic and International, as requested.

3. **Conclusions:** Conversion of DIET facilities to low pressure NET operation is viable and site dependent.

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4. **Contributors:** J. Dobbs

IV. Objective: Support Process Implementation of the Batch NET Process in Bermuda Hundred and BOZ.

A. Strategy:

1. **Results:** Additional conveyance studies were completed at Stockton Street. This was necessary due to the lower bulk density of the NET product. The present system was not able to include 25% NET in MUL. However, it was concluded that this can be remedied by modifications to the system weigh belt.

   Additional tests have confirmed that $5\#/ft^3$ prizing density is reasonable for the NET product. Due to postponement of the Bermuda Hundred project, completion of the NET process and product specifications has been placed on a lower priority.

2. **Plans:** Complete process and product specifications by July. Continue to support Engineering and BOZ operating personnel as requested.

3. **Conclusions:** R&D, Engineering, and Manufacturing personnel continue to operate together as a team with areas that need attention being addressed.

4. **Contributors:** P. Barton, J. Dobbs, J. Tilly, B. Forkins, R. Lum, D. McDowell, M. North, W. Winterson

V. Objective: Support the New Primary Technology Project to Reach 100 mg Weight Reduction.

A. Strategy:

1. **Results:** Close coordination with New Primary Technology and Leaf personnel has resulted in the development of several ET utilization strategies. Several recommendations for expansion conditions for BRICA blends were provided.

2. **Plans:** Continue support as requested.

3. **Contributors:** J. Dobbs, M. Buchanan, M. Hoffman, T. Meade, C. Moogalian, C. Wood

VI. Objective: Develop a Short Cycle Impregnation Process for Use in Small Scale Expansion Systems for PM International.

A. Strategy:

1. **Results:** The dynamic model to simulate the impregnation, as well as the CO_2 supply and recovery system, has been transferred to Richmond and system debug has been started. Four major process configurations have been selected to evaluate the alternative processes with the dynamic model.

   Simons–Eastern Consultants, Inc. has been selected for detailed engineering services to design the plant. Process flow diagrams and P&ID's for the tobacco

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infeed, obloid tower expansion, and reordering systems are near completion. Refining design criteria to meet international standards, codes, and regulations has been initiated.

An indexing table for the tobacco infeed carousel has been selected and detail design of the carousel system has been started.

A preliminary evaluation of a low pressure Panda superheated steam tunnel as an expansion alternative to the tower successfully demonstrated process feasibility. NET filler was expanded to a CV of 9.4 with an equilibrium OV of 11.7, at a Panda exit OV of 4%. Throughput was 120 pounds per hour. Good expansion was obtained at throughputs up to 180 lbs/hr, however, higher exit OV's resulted in lower CV's. A material and energy balance to assess process costs was initiated.

Batch in–situ expansion was evaluated using the Hauni steam tunnel model. The model showed significant top to bottom OV variation would result in batch expansion beds of practical depths. The model predictions were in line with operating experience on the Panda and tower.

2. **Plans:** Operate the dynamic model to evaluate alternative impregnation and CO_2 processes. Select and optimize the impregnation and the best CO_2 supply and recovery processes using the dynamic model. Complete detail design of the tobacco handling system. The impregnator will be placed on order if project funding is approved, and detail design with Simons–Eastern will continue. Equipment arrangement within all modules will be defined. Prepare and implement a process development plan for an alternative tobacco expansion process applying the low pressure, superheated steam tunnel technique.

3. **Conclusions:** The project is progressing well. Continuation will depend on the approval of additional funds in the Second Revised Budget.

4. **Contributors:** P. Barton, P. Chen, K. Cho, J. Dobbs, A. Kumar, D. Leister, R. Lum, W. Nichols, R. Prasad

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PROGRAM NAME : Operations Support
PROGRAM COORD. : R. N. Ferguson
WRITTEN BY : Contributors
PERIOD COVERED : Second Quarter, 1993

Coordinator Summary: Due to the large range of activities in support of various departments the quarterly update will continue to be divided into five sections. These are environmental support, analytical methods applications, support for leaf materials, direct and indirect materials evaluation, and support to finished product quality. Each of these sections is preceded by a brief section summary.

ENVIRONMENTAL SUPPORT

Summary: The installation of the Sly Pilot Scrubber in the BL plant has been completed. Testing is underway to develop scale up data for the scrubber. Testing continues on the Biological Treatment Process. Both steps in the removal and destruction of the components in the BL stack gases continue to perform very well. The project will be completed July 1.

Two processes for phosphine abatement will be pilot tested in July during the warehouse fumigations. The first is the catalytic gas phase oxidation of phosphine using a 0.25 wt. % silver on activated carbon that is currently practiced at the fumigation chamber. The second is a novel liquid phase hydrogen peroxide oxidation process using catalytic amounts of silver nitrate and ferric nitrate. The July testing will develop data to demonstrate the effectiveness and costs associated with both process alternates.

Analytical support of the Environmental program continues. Sampling and analysis on emissions from factory stacks and new primary unit operations were conducted.

I. **Objective:** Develop technology to reduce the levels of various components (nicotine, ammonia, and VOC's) in air emissions at the BL plant to support Environmental Engineering in maintaining compliance at the site and preparing for future permitting requirements.

A. **Strategy:** Evaluate scrubbing technology for treating the air discharges.

1. **Results:** Engineering requested that a Sly Pilot Scrubber be evaluated to determine its performance and develop scale up data. The installation of the scrubber has been completed, and the unit has been started up. This pilot scrubber is treating 2100 CFM of exhaust gas from the South main dryer (the same location that was used in packed bed process development).

   Testing of the unit continues to define the operating performance as follows:

   - Flow rate and optimum feed point of the fresh water. Water flows of 0.6–2.0 GPM at various feed points are being tested.
   - Flow rate and feed location of the recirculated liquor. The testing is being done to improve the operating efficiency of the scrubber.

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- Pilot testing continues on the biological treatment process in the 100 gallon sequential batch reactor. Results continue to be excellent under a variety of operating conditions.

2. **Plans:** Testing of the Sly Scrubber will continue to define optimum conditions for operation of the unit. An extended run is then planned to demonstrate its performance. Testing will be completed by July 1.

   Pilot evaluation of the Biological Treatment Process will continue to develop the following information:

   - Increasing the biomass in the SBR to increase the bio reaction rate thus reducing the size of the basins.
   - Determining the maximum amount of biomass that can be used in the process.
   - Demonstrating the effect of temperature on the reaction rate of the process. Completion of the testing will occur by July 1.

3. **Conclusions:** Scrubbing of the stack gases from the BL plant continues to be a viable alternate for treating these effluents. Biotreatment of the liquor from the scrubber continues to be a viable alternate for destroying the components contained in the liquor.

4. **Contributors:** Reconstituted Tobacco Division and Development Engineering

II. Objective: To support environmental engineering's development of a catalytic carbon process. Evaluate alternate processes that have an economic advantage over the catalytic carbon process.

A. Results and Conclusions:

Analysis of Silver–impregnated Carbon. A virgin sample of Ag/C, previously prepared by Degussa, was analytically evaluated to characterize the material. The X–ray powder diffraction analysis revealed the presence of graphite and calcite. Silver was not observed by X–ray analysis. The SEM analysis indicated the silver was widely distributed in the carbon as discrete particles less than 1 micron in size. The concentration of silver on the carbon was 0.22%.

Phosphine Abatement. After extensive laboratory evaluation of many potential oxidation systems, a recommendation describing the most efficient aqueous chemical treatment system to oxidize/destroy phosphine has been made to C. Bridges (Environmental Engineering) (Memo dated 3/26/93). This system will be used in the July 4 warehouse fumigation study. The specific aqueous solution includes 10% hydrogen peroxide, 0.01 M silver nitrate, and 0.01 M ferric nitrate.

Several commercial portable phosphine monitors have been evaluated. Based on these extensive laboratory evaluations, additional Matheson Gas phosphine monitors will be purchased and used in the July 4th fumigation study.

The secrecy agreement with Johnson and Matthey, concerning conducting research to catalytically oxidize/destroy phosphine, has been completed. Work continues on completing the secrecy agreement with Engelhard.

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1. **Plans:** Conduct the pilot fumigation studies during the July 4th holiday. Continue optimization and larger scale experiments with the recommended system. Evaluate other aqueous oxidant systems. Investigate the synthesis of new supported oxidation catalysts and evaluate their effectiveness. Develop supported oxidation catalysts utilizing the expertise of one or more external companies specializing in catalytic chemistry.

2. **Contributors:** Product Research Division, Analytical Research Division, Environmental Engineering

III. **Objective:** To compare the efficacy of aluminum phosphide, produced by Casa Bernardo, and the typically used magnesium phosphide, supplied by Degesch® in the fumigation of tobacco warehouses and subsequent disposal of the spent materials.

A. **Results and Conclusions:** The experimental and equipment designs are being optimized. This study will compare the two metal phosphides, in a relative sense, for the residual amount of phosphine which can be released from the sachets after fumigation and aeration, and after water deactivation.

1. **Plans:** Complete the construction of the equipment and conduct the study during the July 4th holiday.

2. **Contributors:** Product Research Division, Analytical Research Division

IV. **Objective:** Support the R&D and Engineering five–year plans addressing air, water and solid waste issues.

A. **Strategy:** Provide analytical expertise related to sampling protocols and analytical schemes for qualitative and quantitative descriptions of air, water and solid waste effluents.

1. **Results:**

Effluents. Nicotine was determined by HPLC for the samples taken during the April – May effluent evaluation tests at the 20th Street facility. The analytical data included the appropriate information that is necessary for calculating the quality assurance aspects of the series and were sent to personnel at ERM for inclusion in their final report.

ARD participated in the effluent evaluation tests for the new aftercut cylinders at the Manufacturing Center. Sampling was done by in–house personnel using mini–impingers to determine ethanol emissions.

2. **Contributors:** R. Esperdy, R. McDaniel, B. Ryan, I. Smetena, S. Wahab, S. Yang

ANALYTICAL METHODS APPLICATIONS

Summary: Investigations which were initiated with DBC blend components have progressed to the components of the BRICA blend or the blend itself this quarter. For example, on line moisture studies utilizing BRICA blend components in the Semiworks have progressed well. A moisture and OV

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facility has been installed on T5 to support these studies. The single cigarette blend composition study has moved to BRICA blend components. Laboratory work on the first set of samples is complete and data analysis is in progress. An investigation of the uniformity of burley spray application to BRICA burley tobacco comparing DCC and conventional application was also conducted. The variation of sugar across strips was used to indicate the uniformity of application.

Some potential applications of tin–oxide based sensor arrays were also evaluated this quarter. The Flavor Center must currently use subjective tests to evaluate incoming glycerol for acceptability. A sensor array has demonstrated the potential to differentiate acceptable and unacceptable samples in an initial study. Additional samples will be obtained to determine if these results can be confirmed. Other potential applications of the sensor array technology are also being investigated as part of these studies.

V. **Objective:** Assess current methodologies, develop and implement new technology for moisture determination in tobacco and tobacco materials.

A. **Strategy:** Evaluate the reference method (OV *vs* GC water) used for the calibration of on–line moisture monitors.

1. **Results:** BRICA blend components (bright and oriental) were added to the composite calibration using GC water and the 3–hour OV as the reference methods for the standard algorithm on the Microquad® 8000 (MQ). No significant differences were observed between the DBC strip tobaccos and BRICA tobaccos in the calibration. The MQ has been installed and calibrated using the span and zero derived from the combined regression of off–line evaluations of components encountered at the preblend cylinder. The Koller/Lephardt algorithm has been converted from an absorbance base to an equation compatible with the MQ operation and processing capabilities.

2. **Plans:** Begin off–line evaluation for the final steam cylinder. Start collecting samples in Semiworks.

3. **Contributors:** G. Hicks–White, B. Kanipe, D. Stagg, S. Tenhet

B. **Strategy:** Provide technical support to Cast Leaf personnel for the Moisture Systems Microquad® 8000 sensors and microprocessor.

1. **Results:** The installation of a moisture monitor at exit of C–Dryer has been completed. Training will be provided to Cast Leaf personnel for the general operation, use of cal checks, etc. for the Microquad® 8000 NIR monitors.

2. **Plans:** Assist in transfer of responsibly to cast leaf personnel and assist in training when requested by customer.

3. **Contributors:** G. Hicks–White, M. Parrish

VI. **Objective:** Develop and implement rapid technology for the off–line determination of blend composition.

A. **Strategy:** Develop and implement an analytical tool to perform blend analysis on a single cigarette rod.

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1. **Results:** Initial analysis of the Marlboro samples at target weight and target –30 mg was completed. It was determined in this analysis that utilizing the full spectra and XRF data improved precision. One additional evaluation was completed to determine: 1) the need to use the XRF data at all and 2) the effect of using XRF intensities rather than absolute elemental concentrations. The results showed that for approximately one half of the blend components, the standard errors were smaller when the XRF data were incorporated into the models. Furthermore, the XRF intensities resulted in lower standard errors as compared to the elemental concentrations; the largest difference was noted for DET (~0.40%) absolute at a level of 6.30%. While differences between models using intensities vs concentrations were smaller, using the intensities is preferred because it eliminates the need to calibrate the X–ray spectrometer. The operational plan for the development of the blend analysis system using BRICA blend components has been completed. The first grinding of the BRICA blend calibration mixtures and the set of "pseudo" unknowns have been analyzed by XRF and NIR/Mid IR FT for generating blend prediction models. The model generation using the calibration set data is in progress. The first set of BRICA cigarettes has been analyzed by XRF and NIR/Mid IR FT. A procedure to automate the collection of the NIR/Mid IR FT background spectra has been written and "debugged."

2. **Plans:** Complete NIR/Mid IR FT analysis of the BRICA blend cigarettes and predict component concentrations. Begin development of automated procedure for obtaining IR and integrating IR and XRF spectra from analysis of single cigarettes. Determine feasibility of using Lab Calc software for analyzing combined spectral data.

3. **Contributors:** C. Harward, P. Kurth, B. Martin

4. **Reference:** Harward, C. "Blend Composition Analysis Program Modification," Memo to B. Handy, April 6, 1993.

VII. Objective: Develop and implement monitors for ingredients added to tobacco and tobacco materials.

A. Strategy: Develop and implement a monitor to compare the uniformity of application of burley spray for conventional and new processing techniques.

1. **Results:** BRICA blend burley tobacco strip samples were taken from both the conventional and DCC burley spray application in Semiworks. The samples were analyzed for sugar content using the TM55 NIR sugar monitor. The means and standard deviations of the readings were entered into an RS/1 table and sent to James Gear via computer for data analysis. The uniformity of the sugar application for single strip samples was better using the DCC process compared to the conventional process. The variation of the sugar from strip to strip using the DCC process was as good as the variation observed using the conventional process. The level of sugar applied using the DCC process was less than the level applied using the conventional process and this was confirmed by laboratory results based on ion chromatography (IC). There is no significant difference in the sugar response for

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the top and bottom of the strip samples. Therefore, only one side of a strip sample needs to be measured in future tests.

2. **Plans:** The sugar monitor will be used to verify these findings for a minimum of two more trials in Semiworks. Further analysis is pending completion of the statistical evaluation.

3. **Contributors:** M. Parrish, P. Kurth, R. Pitts, J. Gear

VIII. Objective: Evaluate new technologies for use in off–line, at–line or on–line applications.

A. **Strategy:** Evaluate tin–oxide based sensor arrays for discriminating cigarette filler blend.

1. **Results:** The use of Linear Discriminant Analysis was able to discriminate between gas phase sensor data from representative samples of Marlboro and Merit fillers. The methods of analysis provided a complete breakout of the two fillers when concentration normalized data was used. A more extensive breakout of the data methanol/ethanol/acetone was also obtained using this method. Analysis of data of three glycerol samples obtained from G. Nixon of the Flavor Center (acceptable, borderline acceptable and unacceptable) was completed. An examination of the data indicated that the sensors response to unacceptable and borderline glycerol samples were similar but were always larger than the acceptable sample. Three additional glycerol samples were also tested and the results were similar. Results from these experiments have been presented at a PM Biosenors Task Force meeting and at the PM Analytical Workshop in Milwaukee, WI on May 5 – 7.

2. **Plans:** Consider replicate analyses of glycerol samples. Conduct more analyses of filler and feedstock samples. Begin reimplementation of the piezoelectric crystal sensor technology.

3. **Contributors:** J. Blankinship, A. Das, B. Davies, P. Kurth, B. Martin

SUPPORT FOR LEAF MATERIALS

Summary: The FTR developed procedures have now been integrated into use for crop protection agents at R&D. Improvements in the hplc procedure developed at R&D for Nylar on tobacco are being investigated due to increased interest in this as a possible stored tobacco protection agent. Entomological Services continue to be provided for the replacement of methyl bromide for export cut filler.

IX. Objective: To provide methodology and measurements of crop protection agents (CPA) as needed to insure that tobacco product components and other materials meet regulatory requirements

A. **Strategy:** Establish necessary existing and new methodology in the CPA laboratory.

1. **Results:** Five basic tobacco methods are in place (FTR Part A, organochlorines, Part B, organochlorines and organophosphorus, herbicides, and pendamethalin).

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All initial modifications are now complete and documented. HPLC method for MH–30 has been implemented and evaluation is complete.

NCI techniques by gas chromatography–mass spectrometry using methane reagent gas can confirm approximately 80% of the organochlorine and organphosphorus CPA's. Confirmation of the remainder continues to remain an issue primarily due to low or absent response.

TCLP procedures has been reviewed and laboratory retraining underway. Documentation is approaching completion.

2. **Plans:** Complete performance of the TCLP methods as training exercise and complete documentation by June of 1993. Maintain basic FTR CPA capability. Continue the development of methodology for qualitative and quantitative confirmation of the FTR CPA's at regulatory limits by 12/31/93.

3. **Conclusions:** FTR methodology has been successfully integrated into the R&D environment. Confirmation of the FTR CPA's at regulatory limits continues to appear possible in samples matrices. Methodology for additional CPA's will await input from FTR.

4. **Contributors:** R. Davis, W. McCoy, G. Layman, J. Ware, N. Einolf

B. **Strategy:** Provide documentation of methodology sufficient for laboratory operation and transfer where necessary.

1. **Results:** All applicable FTR methods have been revised and written in R&D format. They are the basis of a R&D CPA procedure manual. TCLP methodology documentation in proper procedure form is virtually complete.

2. **Plans:** Complete documentation for TCLP. Provide documentation of new methodology in R&D format within three months after commissioning. Time line is ongoing.

3. **Contributors:** R. Davis W. McCoy, W. Ryan

X. Objective: To develop an analytical procedure to measure nylar in tobacco.

A. **Strategy:** Finish the development of an HPLC procedure for the determination of nylar in tobacco.

1. **Results:** A preliminary method for nylar in tobacco using reverse phase HPLC has been developed. This procedure is being designed with QA acceptability as a goal.

2. **Plans:** Complete the development of the method in time to support the PRD nylar study.

3. **Contributors:** I. Smetena and S. S. Yang.

4. **References:** Yang, S., Monthly Report, May, 1993.

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XI. Objective: Determine the type of infestation in insect–related customer complaints.

A. Strategy: Examine insect–related customer complaints.

1. **Results:** Three insect–related customer complaints were evaluated to identify the type of insect infestation. Cigarette beetle damage was evident in two of the three complaints. Insect damage in the third complaint was due to cockroaches.

2. **Plans:** Continue to monitor insect–related customer complaints received from Product Audit.

3. **Contributors:** D. Coar

4. **Reference:** Coar, D. L., PM Notebook #9227, p. 39.

XII. Objective: Provide entomological technologies to Philip Morris USA for all aspects of cigarette beetle (CB) control.

A. Strategy: Replace methyl bromide with conditioning to obtain phytosanitary certification of export cut filler.

1. **Results:** Conditioning tests with Oriental were run this month at the Stockton Street (SS) facility. The two tests used 150°F, two and three minute exposure times with 100,000 CBs. CBs have been scored from both test runs and the data showed less than 100% mortality.

2. **Plans:** The conditioning unit at SS facility will be evaluated to assure proper mechanical operation. Additional tests will be conducted with modified test conditions.

3. **Contributors:** T. Burruss, M. Tickle

4. **Reference:** Burruss, T., PM Notebook #8896, pp. 123–124.

XIII. Objective: Determine appropriate methods and evaluate the microflora in tobacco and other pertinent materials.

A. Strategy: Determine the levels of bacteria, mold, and/or yeast in a flavor sample.

1. **Results:** At the request of the Flavor Technology group, a flavor sample was submitted for the enumeration of the indigenous bacterial, mold, and yeast populations. No bacteria or mold were detected in the flavor. However, a significant level of yeasts were detected (as per laboratory standards).

2. **Plans:** Additional flavor samples will be prepared with different formulations with the inclusion of preservative(s). These samples will be enumerated initially and during a predetermined storage period.

3. **Contributors:** D. Chadick, N. Thompson

4. **Reference:** Chadick, D., PM Notebook #9044, p. 115.

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DIRECT AND INDIRECT MATERIALS EVALUATION

Summary: Extensive reviews of the procedures used for either direct or indirect materials have been in progress this quarter. Among the objectives are simplifying the process and reducing the time from sample submission to completion. The focus for laboratory studies continued to be flavors and adhesives. Considerable progress was made on dry flavor procedures. Work was initiated on simplified procedures for major burley spray components. Specialized support was provided for the evaluation of an off specification bright casing.

XIV. Objective: To determine the chemical compositions of materials used or proposed for use in cigarette manufacturing or found in our products.

A. Strategy: Perform appropriate infrared, X–ray, and other analyses in order to determine the chemical composition of the above materials.

1. **Results:** Numerous chemical identifications were made of materials used in the PM manufacturing facilities employing the appropriate analytical procedures. The results and the recommendations were entered into the Materials Evaluation database and reported to Quality Assurance. Materials analyzed included slice tapes, rubber mats, epoxy materials, ear plugs and cords, machine parts, labels, water treatment solutions, inks, adhesives, coating materials, and cleaning solutions. A new set of unauthorized samples was submitted for infrared analysis. Several packs of Marlboro counterfeits from the following countries were sampled including: Vietnam(VIE), Cambodia(CAM), Thailand(TAI), Niger(NIR), Malaysia(MAL), Tahiti(TAH), and Canada(CAN). A summary of these results has been forwarded to the submitter.

2. **Plans:** The activity of the chemical component identification by instrumental methods in support of operation will be continued.

3. **Contributors:** M. Griff, J. Lipscomb, D. Miser, G. Vilcins

XV. Objective: To provide technological support to PM USA for quality improvement by the evaluation or specification of direct and indirect materials. Provide specialized problem solving support.

A. Strategy: Identify the chemical composition of direct materials used in cigarette manufacturing employing several different analytical techniques.

1. **Results:** The analytical work to determine the chemical composition of adhesives used at PM facilities was continued. The procedures used included GC/MS for purge and trap and solvent extracted materials and infrared for the analysis of volatile and non–volatile materials. The program used to determine the lot–to–lot variability of the adhesives has been finished and a report is being written.

2. **Plans:** The results from the program to analyze the adhesives and their lot–to–lot variability will be issued. Other analyses will be performed as necessary.

3. **Contributors:** K. Dudzinski, N. Einolf, M. Griff, C. Keene, J. Lipscomb, K. Sanders, G. Vilcins.

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XVI. Objective: Develop analytical and sensory specifications for incoming flavors used by PM USA. Transfer specifications and methodology to the Flavor Center and Technical Services.

A. **Strategy:** Work with Technical Services, Purchasing, QA, and other R&D staff as required to transfer specifications and methods as vendor agreements are obtained. Discuss specs and methods with vendors to reach agreements on same.

1. **Results:** The flavor specification program was continued. Several specification packages were reviewed, assembled, and forwarded to Regulatory. The vendors' issues were addressed and the packages resubmitted for final vendor signatures. Analytical work on new and revised flavors to generate new specifications was continued. A new odor method utilizing a hot water bath (IMP 32) was written and distributed. As a result of this new method the specifications for glycerine, PG, and isosweet were re–written. Vendor packages for the nine vendors involved were assembled including all methodology pertinent to each vendor and new specifications and submitted to Regulatory. In cooperation with Technical Services Group, re–evaluations were made on the specific gravity and refractive index data from DM codes 01, 03, 04, and 70. The comparison of methods IMP 13 and BDF1 for theobromine, caffeine and vanillin has been completed. The initial results showed that BDF1 is acceptable as a replacement for IMP 13 in cocoa and vanillin containing products. In addition, the comparisons of methods IMP 6 and BDF1 for glycyrrhizic acid in licorice products have been completed and a review of the first results showed that BDF1 can be used in place of IMP 6.

2. **Plans:** Continue to support Purchasing in obtaining agreements with vendors. Develop additional flavor specs as required.

3. **Contributors:** B. Baronian, N. Einolf, K. Sanders, G. Vilcins

XVII. Objective: To identify oxygenated species in heated casing.

A. **Strategy:** To apply specialized mass spectrometry methods to the characterization of materials in heated casing.

1. **Results:** Negative ion FAB analysis revealed the presence of materials which possibly were reaction products between sugars and humectants. Model systems were investigated to confirm this hypothesis.

2. **Conclusions:** Sugars and humectants react to produce secondary products under suitable conditions.

3. **Contributors:** T. Sumpter and N. Jensen

4. **References:** Jensen. N., Sumpter, T., "FAB Mass Spectral Analyses of Bright Casing," Memo to K. Lam, April 23, 1993.

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XVIII. Objective: To identify differences between 'good' and 'bad' Bright casing samples.

A. Strategy: To use 13C NMR to characterize and identify components in Bright casing.

1. **Results:** No differences were detected between the 'good' and 'bad' samples of Bright casing. This implies that the components that make one sample 'bad' is less than one percent of the total mixture, therefore, not detected by this technique, or that they were obscured by the major peaks.

2. **Contributors**: R. Bassfield

SUPPORT OF FINISHED PRODUCT QUALITY

Summary: During the second quarter, extensive resources were utilized for various aspects of product quality. Much of the effort was related to detailed investigation of customer complaints in order to take corrective action, if warranted. In addition, competitive products such as new off price brands and international unauthorized product required a number of analytical procedures for complete evaluation. It is anticipated that this level of effort will not be required during the remainder of the year. However, if these samples continue at the recent level, some other analytical projects may be delayed.

XIX. Objective: To provide methodology for the determination of possible chemical contributions to customer complaint samples.

A. Strategy: Submit customer complaint samples to standard methodology when appropriate. Invoke other analytical resources when necessary to determine the identity and likely source of the responsible chemical components.

1. **Results:** Six customer complaints have been received. Samples CC93014–93016 are in the process of resolution with a series of studies to identify a possible sulfur containing contaminant. Sample CC93017 appears to have the same problem as CC93014–CC93016. Sample CC93018 appears to contain 2,2'–diethyl–1,1'–biphenyl from an as yet unknown source. Sample CC93019 contains 4–methyl–2,4–diphenylpent–1–ene from an unknown source.

2. **Plans:** Attempt identification of contaminant from samples CC93014–CC93016. Continue to provide analytical support for customer complaint issues. Investigate a consolidated data base to facilitate the gathering of information necessary to resolve customer complaint issues.

3. **Contributors:** R. Davis, G. Layman, N. Jensen, T. Sumpter, N. Jensen, B. Baronian, R. Southwick, M. Southwick, L. Hathcock, S. S. Yang

4. **References:**

Davis, R., PM Notebook #9418.

Davis, R. "CC Control 93019, Virginia Slims Ultra Light 100, Flip Top Box," Memo to S. S. Yang, May 12, 1993.

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XX. Objective: To determine if foreign materials are present and to characterize morphology and elemental content of the same in customer complaint samples.

A. Strategy: Use light microscopy, scanning electron microscopy, and energy dispersive spectroscopy to analyze the samples.

1. Results: Six customer complaint samples were examined and are listed below.

- Marlboro Medium cigarettes (#93004–Cox) were collected by Richard Cox on location and were examined by microscopy. No foreign materials were found among the tobacco shreds of the cigarettes. A darkened area, possibly produced by heating or scorching, was observed on the cigarette rod paper.
- One Basic Lights 100's cigarette (#93006) was examined for foreign material and nothing was found.
- Marlboro Lights 100's cigarettes (#93009) were examined in which the customer claimed that a filter plug from one of the cigarettes became lodged in his throat. It was determined that the filter plug had come in contact with epithelial cells similar to those found in the mouth area, but this information did not confirm that the filter plug was lodged in the customer's throat. It also was determined that the adhesive layer on the plug wrap was intact, and that the adhesion was good between the filter plug and the adhesive layer on the plug wrap. In addition, a second cigarette in the pack contained an extra tipping paper wrapped around the filter.
- A brown stain was examined that was on the tipping paper of a cigarette of Parliament Lights HP (#93011). The stained area produced a positive response to the presumptive test for blood as did a control blood sample. Two control blanks gave negative results.
- Sample #93007 contained one smoked cigarette and a match stem. No heavy metals such as those associated with explosive loads were found on the cigarette rod paper. The flare–up claimed by the customer may have been caused by the match stick that was found in the sample.
- Sample #93010 contained a piece of wire in a pack of cigarettes. The metal had an Fe core coated with a Ni layer which was similar in composition to that of a paper clip control.

2. Contributors: V. Baliga, L. H. Thompson

3. References:

Baliga, V., "Customer Complaint 93004–Cox," Memo to S. S. Yang, March 5, 1993.

Baliga, V., "Customer Complaint #93006," Memo to S. S. Yang, March 15, 1993.

Baliga, V., "Customer Complaint #93009," Memo to S. S. Yang, April 16, 1993.

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Baliga, V., "Customer Complaint #93011," Memo to S. S. Yang, April 8, 1993.

Thompson, L. "Customer Complaint #93007," Memo to S. S. Yang, March 29, 1993.

Thompson, L. "Customer Complaint #93010," Memo to S. S. Yang, April 15, 1993.

XXI. Objective: Cigarettes that were made at Cabarrus Manufacturing Facility were examined to determine the nature of lavender spots on the cigarette rod paper.

A. Strategy: Analyses of the spots on the cigarette rod paper were completed using microscopy techniques as well as various mass spectral techniques which included positive and negative ion fast atom bombardment (FAB) and direct probe electron impact (EI) analyses.

1. **Results:** By light microscopy analysis, the spots appeared to have originated from the inside of the rod. However, the spots were not associated with any particular shred type. By mass spectral analyses, it was determined that the principal component of the spots was a hydrocarbon material very similar to a known hydraulic fluid. Polar or tobacco materials were not found to contribute significantly to the spots.

2. **Conclusions:** The spots originated on the inside of the cigarette rod. The spots contained a hydrocarbon material that was very similar to a known hydraulic fluid.

3. **Contributors:** V. Baliga, D. Miser, N. Jensen, T. Sumpter

4. **References:**

Baliga, V., Miser, D., "Spotted Tobacco from Cabarrus," Memo to D. Watson, April 8, 1993.

Jensen, N., Sumpter, T., "Analyses of Lavender Spots on Basic Cigarettes," Memo to G. Crowe, April 5, 1993.

XXII. Objective: To characterize a competitor's product.

A. Strategy: Adhesives and overwrap were analyzed by infrared microscopy, tear tapes and tipping papers were analyzed by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), infrared microscopy, and scanning laser microscopy, and amino acids of the tobacco were analyzed by NDA derivatization and HPLC with florescence detection. Other tobacco analyses included ion chromatography, gas chromatography, liquid chromatography, and colorimetry by the Chemical Analysis Section of ARD, optical microscopy and smoke analyses by Cigarette Testing, smoking by the Richmond panel, and head space analysis.

1. **Results:** The cigarettes resembled Marlboro in appearance but were made of different materials and tobacco. The tipping ink composition and tipping pattern were virtually identical to that used in a Marlboro. However, the tipping base paper

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and cigarette rod paper (wood pulp vs. flax) were different from that of a Marlboro. The tobacco filler probably contained some off–shore tobaccos. It seemed to consist of some oriental, a small amount of burley, probably some bright tobacco, and a component that was probably fermented black tobacco. No flavor additives were detected. The tear tape was of a different construction (surface printed vs. extruded) and different pigment composition than that used in a Marlboro. The tear tape adhesive consisted of poly 2–ethyl–hexylacrylate which is different from that used in our Marlboro product. Packaging materials contained optical brighteners which are not used in U.S. Marlboro. Most of the other materials were not unusual.

2. **Conclusions:** The cigarettes resembled Marlboro in appearance but were made of different materials and tobacco.

3. **Contributors:** V. Baliga, M. Griff, Kai Lam, D. Miser, I. Smetena, S. Yang, the Chemical Analysis Section of ARD, Cigarette Testing, and smoking by the Richmond panel.

4. **References:**

Atkinson–Ballos, D. F. "Richmond Panel Evaluation," Memo to A. C. Lilly, May 11, 1993.

Baliga, V. "Tear Tape Characterization of Sample N3BO," Memo to Don Miser, May 12, 1993.

Chambers, E. "USA King Size Cigarettes," memo to R. A. Fenner, May 13, 1993.

Griff, M. "N93028/N3BO– Infrared Analysis Of U.S.A. Cigarettes," Memo to Don Miser, May 12, 1993.

Miser, D. "Analyses of USA Cigarettes," Memo to C. Lilly, May 13, 1993

Ryan, N. R. "Microscopic Examination of USA Cigarettes," Memo to D. Self, May 13, 1993.

XXIII. Objective: To characterize unauthorized and authorized licensees, affiliates, and competitors products as well as raw materials from known vendors.

A. **Strategy:** Analyze adhesives and overwrap by infrared microscopy, tear tapes and tipping papers by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), infrared microscopy, and scanning laser microscopy, tobacco analyses by ion chromatography, gas chromatography, liquid chromatography, and colorimetry by the Chemical Analysis Section of ARD, and optical microscopy by Cigarette Testing.

1. **Results:** Tahitian Cigarettes

Samples F3–006 TAH and F3–007 TAH were picked up in Tahiti. Markings on the packages suggested that they were US export.

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2. **Conclusions:** Chemical analyses of the tobacco, RL, RCB, ES, ET, and shred length were consistent with a US Marlboro blend and flavoring system. The rod paper filler type, rod paper fiber type, tipping inks, and base sheet compositions, most of the adhesive compositions, pack overwrap compositions, tear tape pigment, tear tape compositions, tear tape outline, filter tow composition, filter fiber denier, and ventilation type, were also consistent with the materials used in US Marlboro.

3. **Contributors:** D. Miser, V. Baliga, M. Griff, Chemical Analysis Section, Cigarette Testing.

4. **Results:** Canadian Cigarettes

   Samples F3–009 CAN and F3–010 CAN were obtained from the Canadian police. Markings on the packages suggested that they were US export.

5. **Conclusions:** Chemical analyses of the tobacco, ES, ET, and shred length were consistent with a US Marlboro blend and flavoring system. The tipping inks and base sheet compositions, all of the adhesive compositions, pack overwrap compositions, tear tape pigment, tear tape compositions, tear tape outline, adhesive applications, filter fiber denier, ventilation, paper permeability, filter RTD, and dimensions of the filters and cigarettes were consistent with the materials used in US Marlboro.

6. **Contributors:** D. Miser, V. Baliga, M. Griff, Chemical Analysis Section, Cigarette Testing.

7. **Results:** Canadian Tobacco

   Approximately 44 pounds of loose tobacco were received from Canada. The tobacco came in the form of 20 samples each of 2 large lots. The lots are marked "Exhibit # 92–222" and "Exhibit # 92–225". These lots have been coded F3–011 (N3AV) and F3–012 (N3AW) respectively. The samples were retained in locked cabinets with restricted access. Sample N3AW was the lighter color of the two tobaccos. Some of the samples of N3AV were noted to have a moldy odor.

   The tobacco blend (oriental, burley, and bright), blend components (expanded and reconstituted tobacco), and flavor systems of the suspect samples were unlike that used in Marlboro or by Philip Morris. The blend used in F3–011 (probably burley, bright, and oriental) was different than that used in F3–012 (burley and bright). Chloride was higher than that used by Philip Morris and higher than that found in domestic tobacco, indicating an off–shore source. Group A organochlorines were very low. Chemical analyses of sample F3–011 suggested that a fermentation process had occurred.

8. **Contributors:** D. Miser, Dick Davis, Chemical Analysis Section, Cigarette Testing.

9. **Results:** Cambodian and Vietnamese Cigarettes

   Sample F3–013 CAM was picked up in Cambodia. The pack appeared similar to others that have been obtained from that region. Sample F3–014 VIE was a sample

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of the brand Lotaba that had been produced in Vietnam. Sample F3–015 VIE was a sample of Champion that had also been produced in Vietnam. Both samples F3–014 and F3–015 were box products and were analyzed for comparison with other samples picked up in Southeast Asia.

The results suggested that the tear tape supplier of all three samples was the same. However, the unauthorized cigarettes were probably not manufactured or packaged by the same operations that produced F3–014 or F3–015. Some similarities in the packaging materials of F3–013 and F3–015 did exist. Tobacco analyses suggested that the blend used to make F3–013 was significantly different than the blends used in either F3–014 (Lotaba) or F3–015 (Champion). Similarly, packaging components suggested a different manufacturer for F3–013 from F3–014. However, the tear tape composition and the pigment of the tear tapes of all three samples were the same. The only other samples examined by this lab with the same tear tape characteristics were those of unauthorized products found in Southeast Asia and are believed to be manufactured by a single operation.

10. **Conclusions:** The results suggested that the tear tape supplier of all three samples was the same. However, the unauthorized cigarettes were probably not manufactured or packaged by the same operations that produced F3–014 or F3–015.

11. **Contributors**: D. Miser, V. Baliga, M. Griff, Chemical Analysis Section.

12. **Results:** Unauthorized Product F3–020

    Five cigarettes of sample F3–020 were submitted for blend analysis, specifically for the presence or absence of Burley. The sample was submitted to the Chemical Analysis Section for alkaloids, reducing sugars, and nitrate analyses. Physical characteristics were measured in microscopy. The results indicated that Burley was not present in any appreciable quantity in the blend. The results were needed in 4.5 hours and were reported in 3.5 hours after receipt of the samples.

13. **Conclusions:** The results indicated that Burley was not present in any appreciable quantity.

14. **Contributors:** D. Miser, Chemical Analysis Section

15. **References:**

Baliga, V., "Tear Tape and Package Gold Pigment Analysis of Fausto Samples F3–005, 006, 007, 008, 009, 010, 013, 014, 015, and Mexico Marlboro," Communication to D. Miser, March 26, 1993.

Miser D., "Preliminary Analyses of Samples F3–006 and F3–007," Memo to G. Morgan, March 3, 1993.

Miser D., "Summary of Analyses of Samples F3–006 and F3–007," Memo to G. Morgan, March 5, 1993.

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Ryan, N. R. "Tobaccos From Canada," Memo to G. Morgan, March 23, 1993.

Miser, D. "Summary of Analyses of Samples F3–009 CAN and F3–010 CAN," Memo to G. Morgan, April 20, 1993.

Miser, D. "Loose Tobaccos from Canada," Memo to G. Morgan, April 8, 1993.

Miser, D. "Summary of analyses of samples F3–013 CAM, F3–014 VIE, and F3–015 VIE," Memo to G. Morgan, April 20, 1993.

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PROGRAM NAME : Summary of Other Programs
WRITTEN BY : P. N. Gauvin
PERIOD COVERED : Second Quarter, 1993

Coordinator Summary: In the Second Quarter 1993, the Computer Applications Group completed the modernization of the TPM Smoking Lab. PCs and UNIX workstations were used to replace obsolete data acquisition systems. The application programs were modified to accommodate the new Cigarette Quality System.

The computers were upgraded in the Materials Evaluation Lab with more memory, a hard disk, and a network connection. A Neural Network model for non–menthol cigarette liking was developed. Gas phase sensors and associated analytical techniques are under development to recognize and differentiate between tobacco blends for process monitoring and other applications. Preliminary results indicate that tin oxide gas phase sensor data may be able to differentiate certain organic compounds.

Work is in progress to implement an enhanced steady–state tracking system for the BL Plant dryer belt and also to develop an automatic startup system for the belt. The investigation of the potential use of pattern recognition technology is in progress for the identification and classification of shredded blend components.

In the Predictive Maintenance Program the development of vibration analysis as a tool for predicting and diagnosing machine health has continued. Sensors were permanently mounted on a packer located in Bay 1 of the MC. Vibration signatures from fourteen locations on the packer were recorded to begin building a database. The utility of cepstra for trend analysis of packer performance is also being investigated.

Under the category of Research on Analytical Methods, a method using two mass spectrometers to characterize the charge transfer reaction process is being investigated. An HPLC method has been developed for the determination of aldicarb and its sulfoxide and sulfone metabolites in tobacco. In addition, an HPLC method has been modified to determine theobromine and glycyrrhizic acid in burley spray.

Efforts are continuing to obtain ISO Guide 25 Accreditation for the Product Testing Lab (Consolidated CTSD/Product Audit Lab). The ISO 25 Team continues to develop a quality manual for the Division. The Product Testing Lab (PTL) Steering Team assessed customer requirements for sample volume and turnaround time versus current PTL instrument and manpower allocation. Results were presented to senior management.

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PROGRAM COORD. : Computer Applications
WRITTEN BY : M. Allred, F. Beers, J. Blankenship, R. Lipps, T. Newby, J. Palesis, J. Stimler
PERIOD COVERED : Second Quarter 1993

I. Objective: Migrate the CTSD Instrument Workstations (IW) to the new Consolidated Laboratories.

A. Strategy: Modify programs on the IWs to handle new bar–codes, sample//test identification, and database load procedures.

1. **Results:** This project is complete. PC and UNIX instrument workstations in the TPM Smoking, Nicotine and Water, Menthol, Material Evaluations and Physical Testing labs are now running on the new Consolidated Lab system.

2. **Plans:** Provide support as needed.

3. **Contributors:** R. Buckner, B. Daughtry, R. Greene, N. Latif

II. Objective: Modernize the TPM Smoking lab.

A. Strategy: Use PCs and UNIX workstations to replace obsolete data acquisition systems in the TPM Smoking Lab. Modify these application programs to accommodate the new Cigarette Quality System.

1. **Results:** This project is complete. The obsolete data acquisition systems have been replaced by a combination of IBM–compatible PCs and UNIX workstations. This was the first implementation of a lab application on UNIX workstations. Although a significant learning curve was encountered, the enhanced, multi–tasking capabilities of the UNIX workstation has provided an effective platform for data collection involving serial communications as well as for lab management. While this project was in progress, the Quality Audit labs were combined with the Cigarette Testing labs. The flexibility of the PC and UNIX computer platforms minimized the effort needed to modify the lab applications to meet the requirements of the new Consolidated Laboratory.

   Key components of this application are manual weighing, robotic weighing, and automated collection of puff counts from manual smokers and from robotic smokers.

2. **Plans:** Implement a Tray Management system. Automate downloading of cigarette lengths. Provide a PC version of the manual weighing station.

3. **Contributors:** R. Greene

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III. **Objective:** Modernize the Materials Evaluation Lab (MEL).

A. **Strategy:** UPgrade the computers in MEL with more memory, a hard disk and a network connection. Automate data transfer between the Firmness and CV/OV instruments in MEL and the CTSD database. Also provide for direct sample handling for Semiworks and NPP. Revise the applications in MEL for the new Consolidated Labs and connect them to the new system.

1. **Results:** This project is complete. Applications in this lab had not been formerly networked. Extensive revisions were required to save results in files, and transfer results to the host database. The scope of the project was expanded to cover modifications for the new Consolidated Labs.

2. **Plans:** Add streamlined sample login for samples from Semiworks and NPP. Transfer raw firmness numbers to the host database so averaging can be done there.

3. **Contributors:** R. Buckner, B. Daughtry, N. Latif

IV. **Objective:** Assist C. Lilly, J. Banyasz, and J. Kao with the development and implementation of a study of possible clathrate formation of nicotine in water.

A. **Strategy:** Obtain data on the solvent water structure in the neighborhood of a nicotine solute with an NPT–ensemble Monte Carlo calculation.

Obtaining the data would use a three phase approach. The first phase would develop a nicotine internal potential model with validation in a single nicotine molecule simulation. The second would develop the nicotine–water potential model with validation in a neat simulation of 128 nicotine molecules. And third, simulation of the binary system of one nicotine in 128 water molecules with data gathering.

1. **Results:** A two hour presentation of the results of the Monte Carlo simulation of nicotine in water was made by myself and J. Banyasz during the Sensory Technology Meeting held April 21st.

2. **Contributors:** C. Lilly, J. Banyasz, and J. Kao.

V. **Objective:** Develop software for the back–propagation training of multilayer feed forward neural networks to eventually replace the existing ANZA Plus neurocomputer and neurosoftware from HNC, Inc.

A. **Strategy:** (1) Develop a C–language program for the back–propagation training and testing of multilayer feed forward neural networks. (2) Prepare a presentation on a software methodology for the automated training and validation of back–propagation neural network models.

1. **Results:** The objectives for this project have been met. Three different C–language programs were developed: (1) "BP Tool" is the basic program for the setup and control of back–propagation learning for a given network of specified configuration, (2) "BP Test" tests the network's predictive performance as a function of hidden layer size, weight initialization, and sigmoid gain, and (3) "BP

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Fold" performs a N–fold cross–validation to estimate the network's predictive performance. These programs implement a modified back–propagation gradient descent algorithm with smoothing and a "derivative offset" in which the learning parameters are automatically adjusted during training.

In an effort to improve computational speed, several enhancements were made to these programs – including the use of register variables and more efficient coding of "dot product" loops. As a result, the performance on pm800 (MIPS processor), pm61 (RS 6000), and pm16 (SPARC 10) substantially exceeds that of the ANZA Plus neurocomputer and even the newer Balboa 860 board from HNC, Inc. For example, a benchmark training run took 68 seconds on the ANZA Plus board but only 11 seconds on a SPARC 10.

The advantages of these C–language programs are manifold: (1) Processing speed on fast processors exceeds that of the ANZA Plus and Balboa boards from HNC. (2) The programs incorporate improved functionality (e.g. an offset to the derivative calculation) which is not available with the ANZA Plus neural network software. As a result, in addition to its faster performance per training cycle, these programs should achieve better mean squared error training and predictive performance in fewer learning cycles. (3) These programs can be used simultaneously by multiple users on varied computing platforms, whereas the ANZA Plus hardware is a single–user system available on only one platform. (4) The $19,000 cost of purchasing a Balboa board (plus annual maintenance) to replace our failing ANZA Plus board is avoided.

A presentation entitled "A Software Methodology for the Automated Training and Validation of Back–propagation Neural Network Models" was presented internally at a R&D Technical Seminar and externally at the Neural Networks Clinic in Troy, Michigan, sponsored by the Society of Manufacturing Engineers in cooperation with the International Neural Network Society.

2. **Plans:** No further work is planned for this objective.

3. **Conclusions:** This software should permit a gradual phase out of the ANZA Plus neurocomputer and neural network software from HNC, Inc. This is particularly timely, because the ANZA Plus hardware is exhibiting signs of age (e.g., corrosion on the connector pins) and is currently failing the Memory Cache diagnostics test which has slowed performance.

VI. Objective: Develop a predictive model for non–menthol cigarette liking.

A. Strategy: Develop a neural network model for non–menthol cigarette liking. Incorporate this model into a defect rating system being developed by Quality Assurance to assess the quality and consistency of cigarette analyticals.

1. **Results:** Employing the newly developed software for back–propagation learning, a three–layer feed forward network was developed to predict non–menthol cigarette liking as a function of the analyticals of the test cigarette and the smoker's regular brand. The model has nine input (explanatory) variables,

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one output variable, and a hidden layer of 11 processing elements. The RMS prediction error as determined by 5–fold cross–validation is 0.38 liking units, which is slightly lower than that obtained for menthol cigarette liking.

2. **Plans:** Incorporate this model into a defect rating system being developed by Quality Assurance, and possibly into a predictive model for PED.

VII. Objective: Develop gas phase sensors and associated analytical techniques to recognize and differentiate between tobacco blends for process monitoring and other applications.

A. Strategy: Develop pattern classification techniques for tin oxide gas phase sensor data in an attempt to differentiate head space gases between tobacco blends.

1. **Results:** Two different sets of data were analyzed by principal component and linear discriminant techniques to assess the ability to classify gas phase sensor data from different organic compounds. The first set of data consisted of measurements of six different tin oxide sensors for organic solvents – 10 samples of methanol, 7 samples of ethanol, and 11 samples of acetone. Each six–dimensional pattern was normalized to unit sum in order to eliminate concentration effects. Linear discriminant analysis of the normalized data showed a clear separation of the patterns between groups. In a leave–1–out cross–validation of classification performance, all but one of the 28 patterns were classified correctly.

   The second set of data consisted of measurements of the same six tin oxide sensors for 8 samples of Marlboro filler and 8 samples of Merit filler. Each six–dimensional pattern was normalized to unit sum in order to eliminate concentration effects. Linear discriminant analysis of the normalized data showed that the sensor patterns for the two groups can be separated by a hyper plane in six–dimensional space – with the 8 Marlboro patterns on one side and the 8 Merit patterns on the other side. In a leave–1–out cross–validation of classification performance, all but two of the 16 patterns were classified correctly.

2. **Plans:** Apply back–propagation and learning vector quantization neural networks for classification and recognition of gas phase sensor patterns.

3. **Conclusions:** Preliminary results indicate that tin oxide gas phase sensor data may be able to differentiate certain organic compounds, including possibly tobacco blends.

VIII. Objective: Laboratory Support of Data Collection and Reporting in the Cigarette Testing Facility

A. Strategy: A computer–based database system was developed to encompass:

- The requests for testing
- The collection of data from laboratory instruments
- Statistical screening of those data
- Reporting of the data

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- Long–term storage of the laboratory data.

1. **Results:** The CQS (Cigarette Quality System) was developed jointly by R&D's CTSD division, QA's Factory Audit facility, R&D's Computer Database Group and R&D's Computer Lab Systems Group. The system is in full use in the laboratory and is currently undergoing feature enhancements for additional "user–friendly" access, and speed increases. In addition, some international data previously tested by CTSD was transferred to Europe. This data is now being received by CAD via Information Systems from Europe, reformatted and sent directly to IOS.

2. **Plans:** Documentation of the system will be prepared to include a process flow document, form layouts, database schemes and general usage instructions. Some method modifications (in relation to the calculation of TAR) are expected and will be addressed as prioritized.

3. **Contributors:** R. N. Lipps

IX. Objective: Laboratory Support of Data Collection and Reporting in the Process Monitoring Laboratory.

A. Strategy: A computer–based database system is being developed to encompass:

- The requests for testing (both from within the laboratory and from R&D requesters directly — e.g., Cast Pilot Plant, Semiworks, NPP, etc).
- The collection of data from laboratory instruments
- Reporting of the data
- Long–term storage of the laboratory data.

1. **Results:** To date, forms for request logging and data editing (based on the CQS System model) have been prototyped and approved by PML staff. Instrumentation in the laboratory is being made "database–conversant" using the same protocol as the CQS system. All instruments in the PML laboratory will be able to process data from both the CQS database system and the PML database system.

2. **Plans:** The basic database access forms will be completed this quarter and customer training will be complete. Upgrades to the Cast Pilot Plant database and the Semiworks database to electronically request testing from the PML laboratory will follow.

3. **Contributors:** R. Lipps, T. Newby

X. Objective: Implement an enhanced steady–state tracking system for the BL Plant dryer belt.

A. Strategy: Work with expert production line operators to define the heuristics of steel belt tracking and use "fuzzy logic" techniques to build an expert system that emulates human belt control.

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1. **Results:** The expert system which has been running the belt on line 2 since September 1992 on a 486 Personal Computer has been ported to an Allen–Bradley PLC–5 installed on line 3. On–line testing has shown that, by comparison to the conventional PID (Proportional Integral Derivative) controller used on that line, the expert system reduces the tail end side–to–side oscillation of the belt by approximately 40 percent while keeping the head end oscillation approximately the same as under PID control. This is a substantial improvement because tail end oscillation is primarily responsible for process component wear and tear. It is also notable that improved tracking is achieved with substantially less "force", i.e., less frequent and smaller control actuator movements. Process component wear and tear is also expected to be reduced as a result of this control force decrease. In view of the improved tracking, BL plant management released the expert system on line 3 for steady–state belt tracking in April 1993.

2. **Plans:** When BL Plant Engineering completes the hardwiring of the necessary process control signals, the expert system will also be installed on lines 1 and 2 using the Allen–Bradley PLC–5 platform.

3. **Conclusions:** It is important to note that the line 3 steel belt has a substantially different tracking signature (determined by belt physical structure) than the line 2 belt. Thus, the fact that the expert system, which was developed on line 2, also achieves superior tracking on line 3 indicates that the fuzzy control strategy it employs does not need to be modified to handle different belts.

4. **Contributors:** J. Palesis, R&D; N. Nunnally, R&D; J. Hickle, BL Plant; J. Davis, Man. Eng.

XI. Objective: Develop an automatic startup system for the BL Plant dryer belt.

A. Strategy: Work with experienced production line operators to define the heuristics of manual belt startup and implement an expert system that uses these heuristics to automatically restart the belt after a track–off occurs.

1. **Results:** For the last three months, the expert system has gone through several testing and refinement stages. During the April BL Plant shutdown, the current version of the expert system was tested extensively on line 2. Well over 20 different tests were conducted with the belt at all likely (and some very unlikely) track–off positions. The expert system automatically restarted the belt with a success rate of 100 percent. The average startup time under expert system control was approximately 6 minutes, i.e., 20 percent of the 30 minute average manual startup time. BL Plant management, who participated in some of the testing, released the expert system for regular use on line 2 in April 1993. Line operators have since used the expert system during regular production to restart the belt. The expert system has maintained a 100 percent success rate.

2. **Plans:** Install the automatic belt startup system on the other two lines as soon as the necessary signal hardwiring is completed.

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3. **Conclusions:** The expert system reduces the average startup time by 80 percent compared to manual startup. As a result, the expert system is also expected to reduce, by the same amount, costs directly related to belt startup after track–off.

4. **Contributors:** J. Palesis, R&D; N. Nunnally, R&D; J. Hickle, BL Plant

XII. Objective: Identify new technologies and techniques with high potential for the solution of business problems.

A. Strategy: Investigate the potential for utilization of pattern recognition technology in the identification and classification of shredded blend components. Develop same with the goal of cost effective implementation as a routine monitoring tool.

1. **Results**: Images of ten shreds of each of the ten current blend components were collected for further study. A texture spectrum calculated from the average of the eight orientations proved to have the same characteristics as an individual one. This provides an orientation free method for calculating texture spectrums.

   The ten average texture spectrums for burley, bright, and Marlboro Turkish were further analyzed to attempt to develop discrimination methods. The 6561 original peaks were first reduced to the 962 peaks containing non–zero frequencies. These 962 peaks contained a large number which were linear combinations of each other allowing a reduction to 64 peaks. A correlation study further reduced the number of peaks to 24 based on high correlation with the dependent variables and relatively low correlation with other independent variables. Multiple regression analysis, considering the three component types as categorical variables, explained 85%, 88%, and 99% of the variation for burley, bright and MT respectively. The simultaneous equations correctly identified each of the shreds as belonging to the correct component type.

2. **Plans:** The above analysis will be extended to the seven component types not yet analyzed. The goal will be to develop a set of regression equations using a minimum of peaks which explain a relatively high percent of the variation and correctly classifies all of the ten component types.

3. **Conclusions:** Image analysis techniques can be used to classify burley, bright and MT shreds in a tertiary system. Based on the texture spectrum approach an orientation free method has been developed to represent a tobacco shred. The 6561 peak texture spectrum can be reduced by 1 or 2 orders of magnitude and used as the basis of component classification.

4. **Contributors:** J. Stimler, G Bokelman, D. McRae

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PROGRAM NAME : Predictive Maintenance
WRITTEN BY : S. N. Ganeriwala
PERIOD COVERED : Second Quarter, 1993

I. Objective: Develop vibration analysis as a tool for predicting and diagnosing machine health and as a design aid for improving machine performance.

A. Strategy: Establish procedures for obtaining vibration signal histories of packers at the Manufacturing Center. Develop analysis procedures using advanced signal processing techniques.

1. **Results:** Sensors were permanently mounted on the 1A1, X–500, packer located in Bay–1 of the MC. A data communication system was designed, fabricated, and tested for the transfer of multiple signals simultaneously from all sensors on the 1A1 to a TEAC tape recorder. Vibration signatures from fourteen locations on the 1A1 packer were recorded to begin building a database.

   Spectra and cepstra were computed from vibration signatures acquired on packers located in Bay–1 and Bay–4. The driving function for the reciprocating motion was more directly obtained from the cepstrum than the spectrum. The utility of cepstrum for trend analysis of packer performance will be investigated.

   The X–2 packer 4D5 located in Bay–4 packer developed a problem in the Tucker gearbox. The spectrum of the vibration signature signal obtained on the Tucker gearbox indicated the possibility of a bearing type defect. The cepstrum did not provide any additional insight. The vibration signature of a point slightly removed from the Tucker gearbox was entirely different indicating the need to better understand the effect on the signal resulting from different vibration transmission paths.

   The database architecture has been developed using SKF software and the first database signals collected and stored. A similar architecture is being developed on the TEAC tape recorder and signal analysis software package.

2. **Plans:** The accelerometer installation in both Bay–1 and Bay–4 will continue. Vibration signatures will be acquired weekly on the 1A1 X–500 packer in the development of an operational trend and history. Faults will be introduced in the packer subassembly, first wheel knee, located in the Vibration Laboratory in the development of a catalogue of the fault signatures.

3. **Contributors:** S. N. Ganeriwala, H. M. Dante, A. T. Hill

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PROGRAM NAME : Research on Analytical Methods
PROGRAM COORD. : P. Gauvin
WRITTEN BY : N. Jensen and S. Yang
PERIOD COVERED : Second Quarter, 1993

I. Objective: Gas phase reactions are of fundamental interest in smoke chemistry. Many types of reactions and decompositions have been characterized. However, one type of reaction, the charge transfer process, is very difficult to characterize by conventional means. The objective of this work is to develop procedures for performing charge–reversal experiments with the tandem mass spectrometer and to explore these processes in model systems.

A. Strategy: To operate the two mass spectrometers of the tandem system in opposite polarities to facilitate information capture on chemical processes related to charge stripping and charge exchange reactions.

1. **Results:** Charge exchange spectra have been obtained for a number of model compounds including humectants, sugars, and fatty acids. This is most interesting information as the process is fairly facile in some cases but virtually impossible to observe by traditional methods. Evaluation of the products of these reactions is in progress.

2. **Plans:** To pursue investigation of fundamental experiments in this area and explore possible implications regarding smoke chemistry.

3. **Conclusions:** Work is in progress.

4. **Contributors:** N. Jensen, T. Sumpter

II. Objective: Determination of Aldicarb, Aldicarb Sulfoxide and Aldicarb Sulfone in Tobacco Using HPLC with Dual Post–column Reaction and Fluorescence Detection

A. Strategy:

1. **Results:** An HPLC procedure is developed for the determination of aldicarb and its sulfoxide and sulfone metabolites in tobacco. Sample preparation consisted of a single extraction of tobacco samples by methanol with the aid of sonication at ambient temperature for 30 minutes. The extract was filtered and then injected directly into an HPLC equipped with a dual post–column reaction system and a fluorescence detector.

   Chromatographic separation was performed on a C–18 column with a mixture of methanol–water as the mobile phase. Aldicarb residues in the eluate were hydrolyzed in the first post–column reactor with concentrated alkalis at an elevated temperature to yield methylamine and subsequently derivatized in the second post–column reactor with o–phthalaldehyde prior to fluorescence detection (rf: Krause, Anal. Abstr., 1981, 40, 5G41). Recoveries of aldicarb residues from tobacco samples spiked at 5–10 ppm levels range from 80 to 90%.

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2. **Contributors:** S. S. Yang, I. Smetena

III. **Objective:** Analytical Method for the Determination of Theobromine and Glycyrrhizic Acid in Burley Spray Solution

A. **Strategy:**

1. **Results:** An HPLC procedure is modified for the determination of theobromine and glycyrrhizic acid in Burley Spray. Sample preparation consists of a single extraction and has been tested repeatedly to ensure that a representative sample with reasonable size is taken from the inhomogeneous suspension of Burley Spray. Each bottle of Burley Spray, which was submitted twice per week from facilities in Manufacturing Center, was analyzed with 5 replicates. The data show consistent results. Chromatographic separation was performed on a C–18 column using the parameters described in an existing method (BDF Method– 1 for blend dry flavor).

2. **Contributors:** I. Smetena, S. S. Yang

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PROGRAM NAME : PTL ISO 25 Accreditation
WRITTEN BY : J. M. Garman
PERIOD COVERED : Second Quarter 1993

I. Objective: To pursue and obtain ISO Guide 25 accreditation for the Product Testing Laboratory (consolidated CTSD/Product Audit laboratory).

A. Strategy:

- Coordinate with PME, IOS and PM Standards Lab to implement the principles and practices as outlined in ISO 25 and ISOI 9000.
- Develop a quality manual for Product Testing Laboratory.
- Initiate an internal audit system to assess conformance to the quality system.
- Retain a consultant to assess the quality system and its effectiveness.
- Apply for accreditation.

1. **Results:** The ISO 25 team continues to develop a quality manual for the division. As the division is still in transition due to the March consolidation, completion of the quality manual has taken longer than expected. Project teams continue to be chartered to develop documentation for specific analyses and processes. Documentation, requested from support groups (CAD, Building Administration, and PM Standards Lab), has yet to be supplied.

   The Product Testing Laboratory Steering Team assessed customer requirements for sample volume and turnaround time versus current PTL instrument and personpower allocation. The results of this assessment were presented to Senior Management.

2. **Plans:**
   - Complete quality manual.
   - Develop process map of the division.
   - Continue charting project teams to develop documentation of analyses and processes

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